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Limestone-Calcined Clay (LC2) as a supplementary cementitious material for concrete

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Abstract

In this work, limestone-calcined clay (LC2) is studied as an alternative supplementary cementitious material (SCM), combining two widely available resources – calcinated kaolinitic clay and limestone, to partially substitute portland clinker. The primary goal is to assess the potential of LC2 to produce moderate to high strength concretes with design compressive strengths of 20 to 50 MPa. For this purpose, 27 mixes with LC2 were prepared with a range of binder contents and water-binder ratios, and the performance was benchmarked against those of mixes having fly ash (PFA). In addition to the quantification of strength and concrete resistivity, life cycle assessment was performed for the concretes considering a typical situation in India. The efficiency of concretes made with LC2, PFA and ordinary portland cement (OPC) was analyzed using the energy intensity index (ei_{cs}) and apathy index (A-index) as sustainability indicators. This framework establishes the sustainability potential of the LC2 with insights on the influence of strength on the indicators. It is concluded that the LC2 concretes with 45% replacement level, w/b≤0.45 and binder content lower than 400 kg/m^3 possess the highest sustainability potential, among the concretes studied here.

Keywords: Limestone calcined clay (LC2); Low carbon binders; Durability; Life cycle assessment; Sustainability

1 Introduction

Evolving eco-friendly solutions with enhanced concrete performance are primary to the continued use of concrete as a construction material, more so due to the binding material being mainly composed of cement, which contributes to about 5% of global anthropogenic CO_2 emissions [1]. The consumption of cement in India and other emerging economies is projected to increase in the next few decades due to the steady demand for infrastructure and housing. Consequentially, any improvement in terms of the sustainability of the concrete would make a significant impact. Industrial by-products or residues as cement substitutes, such as fly ash and slag, commonly known as supplementary cementitious materials (SCMs), have led to better performance of the concrete, such as higher long-term strength and lower ingress of external chemical species, thereby prolonging the service lives of reinforced concrete structures. However, the global availability of fly ash and slag is expected to reduce over the next few decades due to shifts in the sources of power generation and steel processing techniques, respectively. Recent studies have shown that a combination of limestone and calcined clay has the potential to increase the clinker substitution level to nearly 50% [2-5]. In India, considerable progress has been made in the last ten years in a collaborative research project on 'Low Carbon Cement' involving IIT Madras, IIT Delhi, IIT Bombay and Developmental Alternatives, New Delhi. The project promotes industry uptake and rapid standardization by assessing the technical feasibility of the composite limestone calcined clay cement (LC3), recently standardized as Portland Calcined Clay Limestone Cement (IS 18189) [6].

In this work, a mixture of limestone and calcined clay, called LC2, is assessed as a mineral admixture or SCM to be used directly in concrete production [7, 8], as proposed in the Low Carbon Cement project as early as 2015. For preparing LC2, the kaolinitic clays are calcined at 600-800 °C and blended with crushed limestone. This study reports the viability of using LC2 for producing moderate to high strength concretes. Two performance parameters obtained experimentally in this study are: i) the strength development in the concretes, and ii) the surface resistivity as a measure of durability. Results obtained from the experimental study are used to analyse the sustainability potential of the concrete systems with LC2 and pulverized fuel ash or fly ash (PFA), through a recently proposed framework based on energy intensity and apathy indices that account for carbon footprint and durability ü9]. The implications of the concrete strength on the sustainability potential are also discussed.

2 Research programme

2.1 Materials

A commercially available ordinary portland cement (OPC, 53 grade with 65% alite content, conforming to IS 269 [10]) was mixed with siliceous (ASTM Class F) fly ash (denoted as PFA)

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and limestone-calcined clay as admixtures at 30% and 45% replacement levels, respectively; the replacement levels chosen are the maximum values, as determined previously in laboratory and field studies. The LC2 used here was produced by intergrinding 65% calcined clay, 33% limestone and 2% gypsum, on an industrial scale [11].

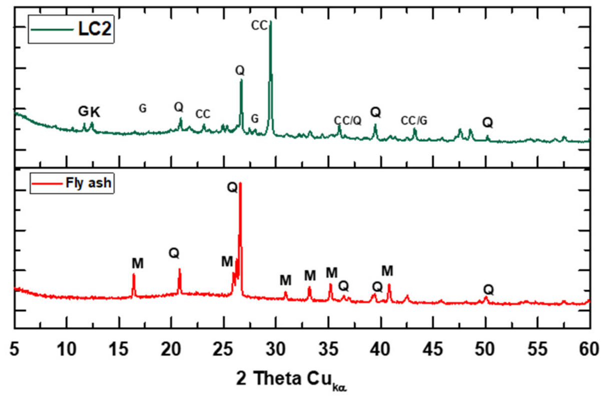


Figure 1. X-ray diffractogram of LC2 and fly ash used in the study. Reproduced with permission from Vaasudevaa et al. [8].

Figure 1 shows X-ray diffractograms indicating the mineralogical composition of fly ash and LC2; the quantitative analysis indicates that the fly ash had about 50% amorphous content and LC2 had 45% amorphous content with 29% calcite. The chemical composition of the materials can be found elsewhere [8]. The major oxides in LC2 were CaO (27.8%), SiO_2 (33.7%), Al_2O_3 (19.1%), with a loss of ignition of 12.4 % from limestone decomposition. The CaO content (from XRF composition) in LC2 is mainly due to limestone that was added to the calcined clay, after calcination. Kaolinitic clay used in the study did not contain any carbonate as impurities (i.e., limestone or dolomite). Hence, no CaO was produced during the calcination of the clay. A laser particle size analyzer was used for characterizing the particle size distributions (see Figure 2); it can be seen that LC2 has a high amount of fines, with the median grain size, d_{50}, being 17 µm and 13 µm for PFA and LC2, respectively, being similar to that of OPC, which is 18 µm.

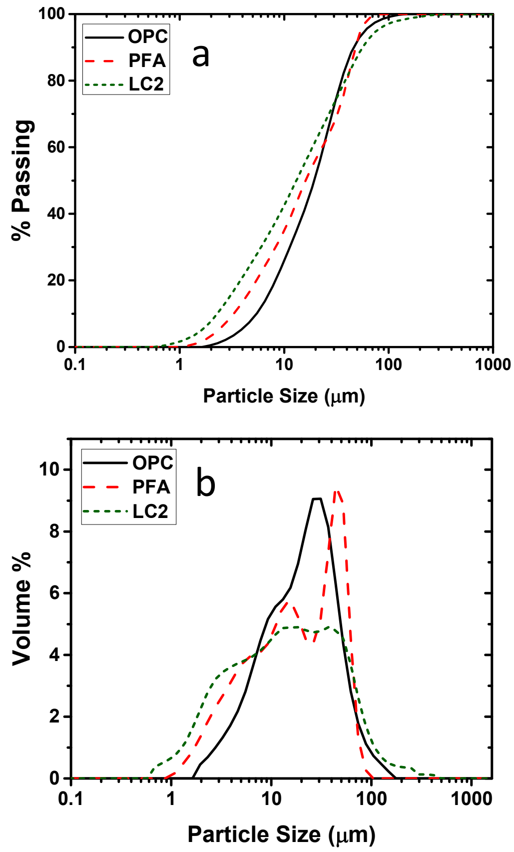


Figure 2. Particle size distributions of PFA, LC2 and OPC used in the study.

An experimental matrix was used to analyze the influence of mixture proportions on the concrete performance by varying the binder content and water-binder ratio (w/b). Table 1 summarizes the 50 mixes studied (marked with ✔) – 27 concretes with LC2 and 23 concretes with PFA. Crushed granite and graded river sand were used as the coarse and fine aggregates, respectively, in the ratio of 55:45, with the 20 mm down (i.e., passing through the 20 mm sieve) and the 10 mm down fractions being proportioned as 55:45 within the coarse aggregate. A PCE-based high range water reducer was used to obtain slumps in the range of 80 to 160 mm, which is considered to accommodate concretes with high (i.e., higher binder content and w/b) and low paste contents. Notably, fly ash mixes required lower superplasticiser dosages than the LC2 mixes; the maximum dosages of superplasticizer were 0.33% and 1.18%, as solid weight to that of the binder, for the PFA and LC2 concretes, respectively.

Table 1. Binder contents and water-binder ratios for the concretes fabricated

Binder type	Binder content (kg/m³)	water-binder ratio 0.35	0.40	0.45	0.50	0.55	0.60
LC2 (55% OPC + 45% LC2)	280				✔	✔	✔
	310	✔	✔	✔	✔	✔	✔
	360	✔	✔	✔	✔	✔	✔
	400	✔	✔	✔	✔	✔	✔
	450	✔	✔	✔	✔	✔	✔
PFA (70% OPC + 30% fly ash)	280				✔	✔	✔
	310		✔		✔	✔	✔
	360	✔	✔	✔	✔	✔	✔
	400	✔	✔	✔	✔	✔	✔
	450	✔	✔	✔			

2.2 Assessment of concrete performance

Compressive strength was measured on 100 mm cubes cured in a mist room till the age of testing; 3 specimens were tested at each of the ages of 3, 7, 28 and 90 days, in a 3 MN capacity system, in accordance with IS 516 [12].

Surface resistivity was measured on a cylindrical specimen of 100 mm diameter using the Wenner four-probe resistivity technique, as per the guidelines of AASHTO T358 [13]. This parameter indicates the ability of concrete to withstand/resist the transfer of ions when subjected to an electrical field. Previous studies have established the relationship between the resistivity and chloride penetrability, with reasonable accuracy [14]. Table 3 shows the classification of concrete quality based on surface resistivity, as suggested by ACI 222R [15] and AASHTO T358 [13], in terms of corrosion rate and risk of chloride ingress, respectively. Since resistivity measurements are sensitive to the surface condition of the concrete, including the presence of moisture and voids, 27 measurements were made on three specimens, for each case, to obtain a representative average. Note that the value provided in Table 2 can be influenced by specimen geometry. The AASHTO recommendation specifies 100 × 200 mm cylinders, which were specimens used in this study. In case of different specimen geometries being used, a suitable geometric correction factor needs to be adopted, as suggested in [16] while using the recommendation in Table 2.

Table 2. Classification of concrete based on surface resistivity

ACI 222-R [15]		AASHTO T358 [13]	
Resistivity (k.ohm-cm)	**Corrosion rate classification**	**Resistivity (k.ohm-cm)**	**Risk of chloride ingress**
<5	Very high	<12	High
5-10	High	12-21	Moderate
10-20	Low to moderate	21-37	Low
>20	Low	37-254	Very low

3 Sustainability assessment

3.1 Methodology and case parameters

Sustainability assessment of concrete is often performed by estimating the environmental impact due to the production of concrete [17,18]. Recently, however, the analysis of sustainability potential has incorporated mechanical and durability performance, with specific relevance to concretes containing SCMs. Consequently, indices have been formulated based on performance parameters, such as strength and durability or service life, to provide a holistic assessment of concrete systems [9, 19-23].

The common technique for quantifying the environmental impact of concrete is life cycle assessment (LCA), as defined in ISO 14040 and 14044 [24, 25], including aspects such as resource consumption, water-depletion, energy consumed, emissions during the production process, upstream cycles (i.e., extraction of raw materials, transportation) and final disposal (i.e., waste, recycling). In simple terms, the LCA methodology involves four main steps: i) goal and scope definition, ii) life cycle inventory analysis, iii) life cycle impact assessment and iv) interpretation.

In this study, LCA was used to calculate the environmental impact in terms of the CO_2 emissions and energy consumed, for different concrete systems with PFA and LC2, as given in Table 1 using the 'ab initio' framework proposed by Basavaraj and Gettu [26]. The ground-to-gate (or cradle-to-gate) system has been adopted for the analysis [17,18]. Primary data for the cement production was collected from an integrated cement plant in South India, near Nandyal, and secondary sources such as ecoinvent, EPA and IPCC were used for complementary data. For the emissions and energy calculation, all the processes in the extraction of raw materials (e.g., limestone, clay) and fuels (e.g., coal, pet coke), transportation of these materials, and electricity production for cement manufacturing process were considered. OPC and LC2 are both considered to be made in the same plant at Nandyal and transported (about 400 km) to the RMC plant in Chennai. Coarse and fine aggregates are to be sourced from quarries that are 75 kms and 192 kms away, respectively. Fly ash was treated as waste and, consequently, only the impact due to transportation from a thermal plant situated 35 km away was considered. The energy required to calcine the clay could vary from 1.9 MJ/kg to 3 MJ/kg depending on the type of calciner used [27]. Here, the calcination energy for the clay was taken as 2.6 MJ/kg based on TGA (Thermogravimetric Analysis) and DSC (Differential Scanning Calorimetry) analysis, and some industry experience. In addition to the calcination process, the emissions and energy consumed by LC2 binder include that during extraction and transportation of clay, fuels and limestone. It should be noted that the energy consumed, and CO_2 emissions of LC2 could vary based on the clay calcination energy and process used in the actual scenario.

3.2 Sustainability indices based on performance indicators

The analysis of the sustainability potential of the different concrete mix proportions considered here employs the indices and decision framework proposed previously in terms of energy consumed and CO_2 emissions [9]. The approach quantifies the environmental impacts, and combines them with performance characteristics, such as compressive strength and chloride diffusion coefficient, to obtain the indicators listed in Table 3.

Table 3. Sustainability indicators used

Index	Formula	References
Energy intensity ei_{cs} $(MJ/m^3/MPa)$	$\frac{Energy\ Consumed}{Compressive\ strength}$	[9, 28]
A-index Ai_{chlor}	$\frac{CO_2\ emissions\ per\ m^3\ of\ concrete}{F_{Chlor}}$ $F_{Chlor} = \exp(10^{-6}/\sqrt{D_{cl}})$	[9]

The first index is the energy intensity (ei_{cs}), which represents the energy embodied in one m^3 concrete per unit compressive strength. Secondly, the apathy index combines CO_2 emissions attributed to producing one m^3 concrete with the durability parameter, $F_{Chlor} = \exp(10^{-6}/\sqrt{D_{cl}})$, where D_{cl} is the diffusion coefficient (in m^2/s) of concrete. The factor F_{chlor} is taken as an indicator of service life limited by chloride attack, as identified in Gettu et al. [9].

For determining the values of F_{chlor}, the concrete resistivity was converted to non-steady state migration coefficient using the relationship presented in Figure 3. This is based on data from of 76 sets of concrete tested at the ages of 28, 90, 180 and/or 365 days in extensive programmes carried out at IIT Madras [3, 29-32].

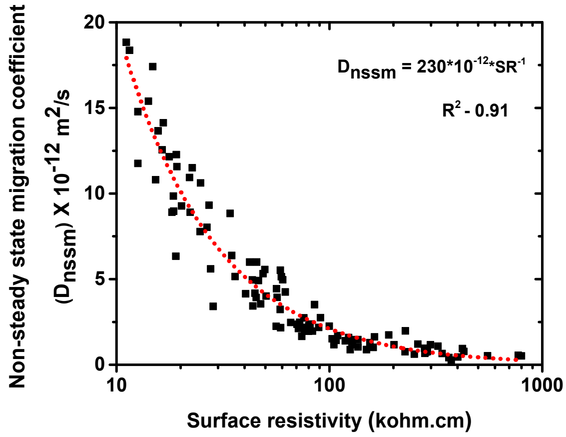


Figure 3. Relationship between surface resistivity (SR) and non-steady state migration coefficient based on previous studies at IIT Madras.

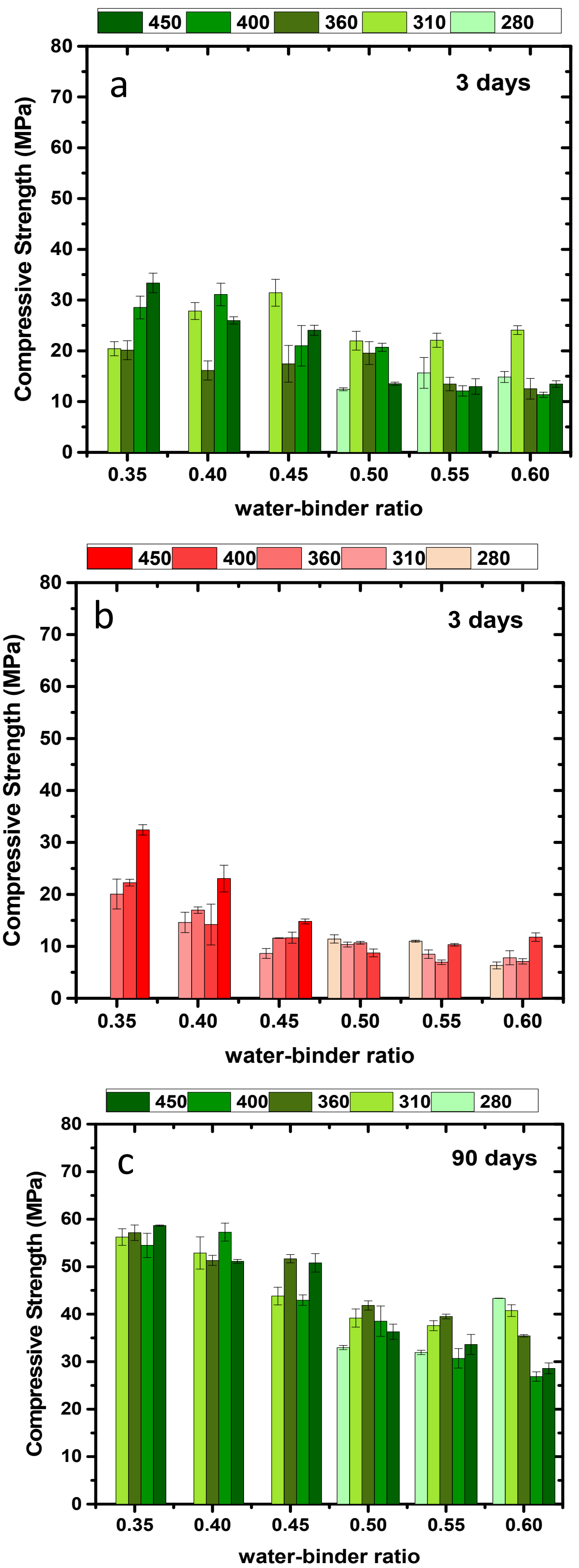
4 Results and Discussion

4.1 Concrete properties

4.1.1 Strength development

For the mainstreaming of LC2 as an admixture in concrete, the ability to yield compressive strengths comparable to more conventional SCMs is essential. Figure 4 presents the compressive strength as a function of w/b for LC2 and PFA concretes at early and later ages, i.e., 3 and 90 days, respectively. Concretes with LC2 have strengths comparable to PFA, despite having 15% lower clinker content (see Figures 4(a) and (b)). In general, the tests on both PFA and LC2 concretes confirmed the dominant role of w/b on the strengths at both early and later ages.

All concrete mixes with LC2 surpassed the 3-day strength of 10 MPa whereas PFA concretes prepared with w/b above 0.5 (i.e., 12 out of 23 concretes reported in the study here) could not. This is in accordance with the expected lowering of the early-age strength development due to the delayed contribution of fly ashes [33]. Two major reasons leading to such a trend are: i) the dilution of the hydrates at an early age and ii) the increase in paste volume at a constant w/b with higher binder content. The former phenomenon leads to higher free capillary porosity that delays the densification of the matrix by the hydration products. However, the PFA concretes with w/b < 0.45 exhibited higher strength with an increase in binder content, as the dilution effect is not significant in such cases. In LC2 concretes, both limestone and calcined clay intervene in the hydration process in a complementary manner at an early age, unlike in PFA. However, besides the improved reaction kinetics, the early age strength of LC2 can be influenced by the higher clinker replacement (nearly 50%) and high superplasticiser demand, both of which could lead to some of the differences at the early age strength across binder contents.



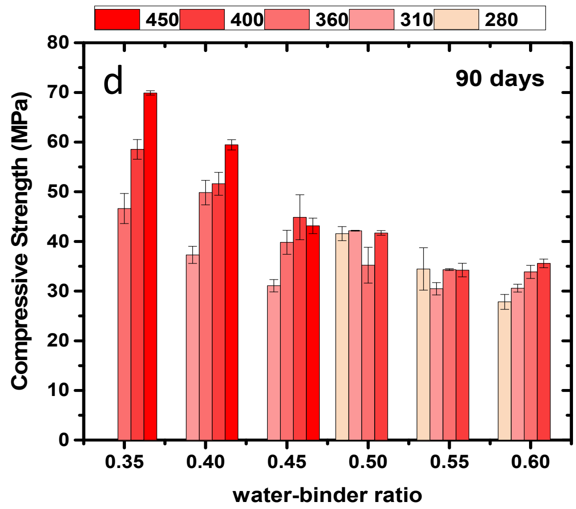


Figure 4. Compressive strength of concretes at 3 and 90 days: (a) LC2 at 3 days, (b) PFA at 3 days, (c) LC2 at 90 days, (d) PFA at 90 days.

Figures 4 (c) and (d) represent the 90-day compressive strengths of LC2 and PFA concretes for various w/b, which decrease as the w/b goes up from 0.35 to 0.60. Notably, the strengths of all LC2 concretes with w/b ≤0.45 surpassed 40 MPa whereas in the case of PFA, only the concretes with a minimum of 360 kg/m^3 of binder and w/b < 0.4 could attain strengths of at least 40 MPa. The results also show that LC2 concretes could attain similar 28-day compressive strengths at marginally higher w/b compared to those with PFA. PFA concretes show a better response to an increase in binder content than LC2 concretes, which could be due to the higher clinker content in the former (30%), as well as limited evolution in the microstructure of calcined clay-limestone systems due to the highly refined pore structure that forms at an early age [3, 30, 34].

The evolution of strength from 3 to 90 days in the concretes with 400 kg/m^3 binder content is shown in Figures 5(a) and 5(b). As discussed earlier, the LC2 concretes had similar strength or better at 90 days than the corresponding PFA concretes. Moreover, the LC2 concrete with w/b = 0.5 was able to reach 20 MPa strength at 3 days, whereas the corresponding PFA concretes attained only 10 MPa and their strength surpassed 20 MPa only at the age of about 28 days.

Figure 5(c) presents the range of compressive strengths obtained in the LC2 and PFA concretes, where the higher strengths of LC2 concrete at 3 and 7 days are evident. The strength band of LC2 is consistently higher than that of the PFA concretes, which indicates the usefulness of the former concretes in structural applications with early strength requirements or situations where extended curing cannot be provided.

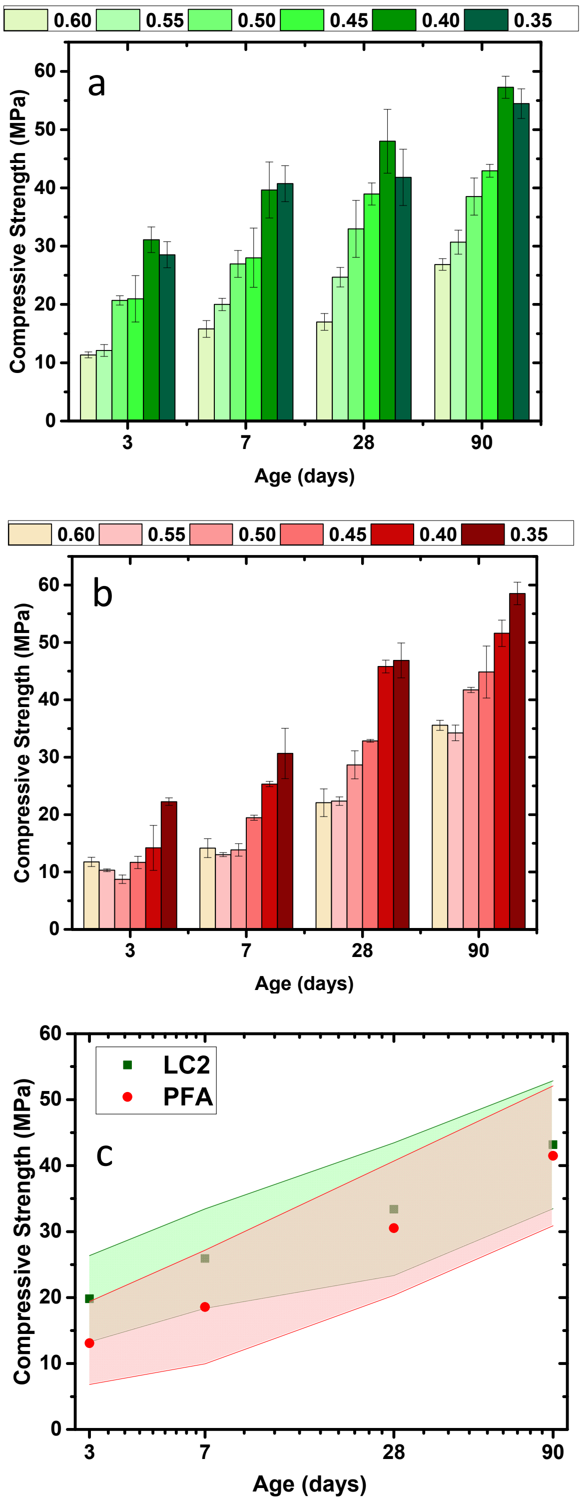


Figure 5. Strength development in concretes with binder content of 400 kg/m^3 having (a) LC2 with binder content of 400 kg/m^3, and (b) PFA with binder content of 400 kg/m^3; and (c) strength development in all the concretes considered in the study

4.1.2 Evolution of resistivity

Figure 6 shows that the surface resistivity of LC2 and PFA concretes increases with curing duration due to the extended pozzolanic reactions from calcined clay and fly ash. The absolute resistivity values of LC2 concrete are always higher than those of PFA mixes at all ages, confirming the trends found in previous studies on cement paste [30, 35, 36] that were attributed to significant modification of the pore structure [36, 37] and pore solution conductivity [30, 36]. Based on the data presented in Figure 6, it is seen that the surface resistivity is more dependent on the binder type than the water-binder ratio, at any specific age, though there is a small reduction with an increasing w/b. However, such reduction due to the change in w/b is insignificant in comparison to that due to the change of binder from LC2 to PFA. Such differences in concrete resistivity between LC2 and PFA concretes are due to the nature of the pore network or the interconnectivity of the pores, which is primarily controlled by hydration products and the denseness of the concrete microstructure [29]. For all concretes, the resistivity exhibits a marked increase between 3 and 7 days for the LC2 concretes, as observed in Figure 6.

The range of resistivity values for LC2 concretes is 50-250 kohm.cm at 28 days, which corresponds to a very low risk of chloride ingress as per AASHTO T 358 [13] (See Table 2). On the contrary, PFA concretes had a resistivity range of 10-50 kohm.cm that reflects a moderate risk of chloride ingress. Similarly, LC2 concretes can be classified to have a low corrosion rate as per ACI 222R, whereas PFA concretes would have low to moderate corrosion rates. Although the resistivity values are significantly higher in LC2 concrete, this could be influenced by pore solution conductivity to some extent. The effect of pore solution can be decoupled using a normalised resistivity of concrete considering the pore solution, as discussed elsewhere [30, 36].

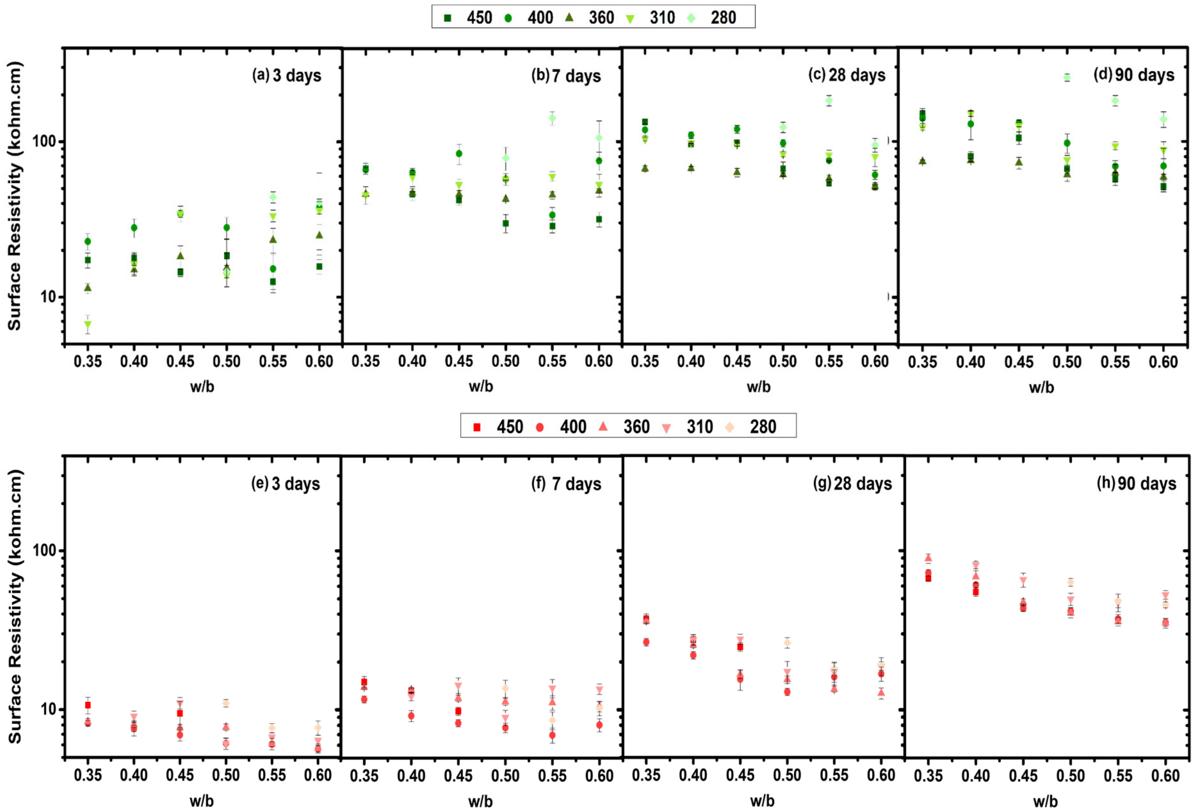


Figure 6. Surface resistivity of concretes with LC2 and PFA.

4.2 Life Cycle Assessment (LCA)

The total CO_2 emissions and energy consumed in the production OPC and LC2 have been summarized in Table 4. These impacts were calculated based on data collected from the cement plants and following the LCA methodology, as explained in Section 3.1. It is evident that LC2 has a much lower impact than OPC, which reflects the benefit of substituting OPC with LC2 in concrete. The corresponding values for PFA are zero since it is considered as a waste, which results in concretes with PFA having lower impact than those with LC2 when the former is available nearby or high replacement levels are required.

Table 4. Results from environmental impact assessment of the binder components

Binder	Emissions (kg CO_2/tonne)	Energy demand (MJ/tonne)
OPC	930	5945
LC2	260	3500

The energy and emissions from the binders can be used along with other relevant data to calculate the impacts of the different concretes considered in Table 1. The contributions from all components in concrete systems, such as binders, water and aggregate, together comprise the total impact. Figures 7(a) and (b) present the energy consumed and CO_2 emissions of the 50 mixes studied here, along with 3 PFA mixes and 15 OPC mixes, from previously published work [3, 29, 33] at the same laboratory. It is evident that the environmental impact of concrete varies significantly with the volume and type of binder, and is not affected by the w/b. Further, as the binder content increases, the impact is higher, as expected.

Both energy consumed and emissions are highest in the case of OPC concretes while PFA and LC2 concretes are found to have similar emissions for concrete at similar binder contents. However, the energy consumed is significantly higher for LC2 concretes in comparison to PFA concretes, due to the energy requirement for the clay calcination process, though not as much as the OPC clinker.

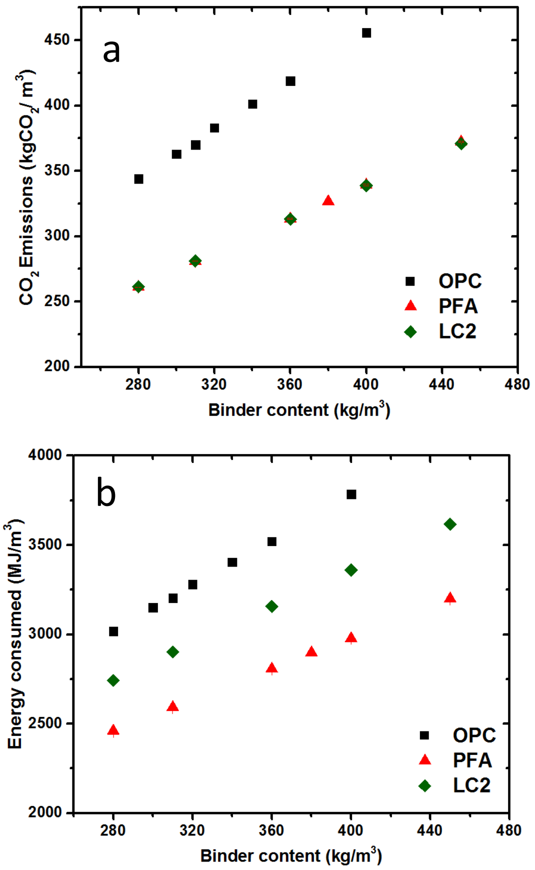


Figure 7. Energy and CO_2 emissions of different concretes used in the study.

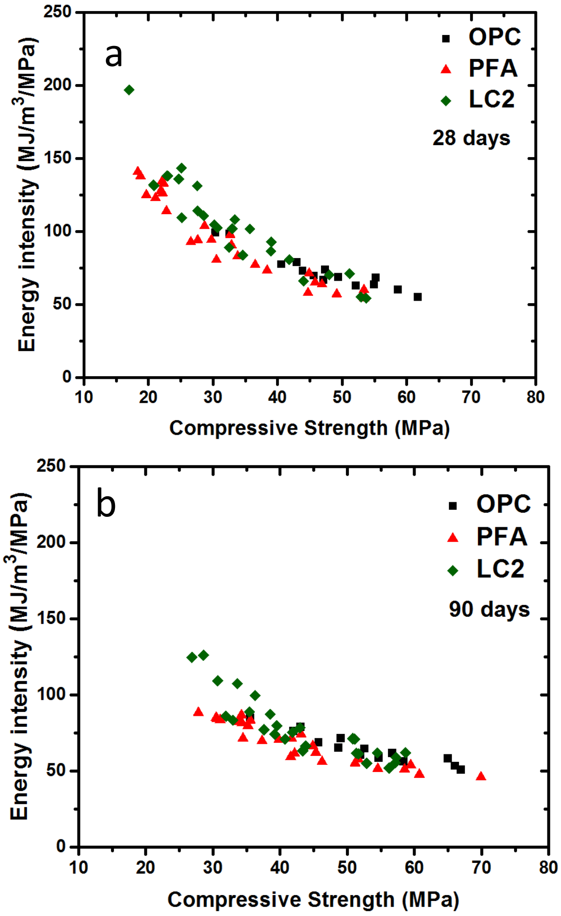


Figure 8. Variation of energy intensity indices with compressive strength, for different binders.

4.3 Energy intensity index (ei_{cs})

Energy intensity index is an indicator used in the sustainability decision framework considered here, which combines the embodied energy with the mechanical integrity represented by the compressive strength. In Figure 8, ei_{cs} is plotted with the compressive strength at 28 and 90 days, where it is seen that, irrespective of the binder type, ei_{cs} decreases with an increase in strength. For the concretes in this study, the ei_{cs} range is similar to that reported previously [9]. The least ei_{cs} observed is 47.8 ($MJ/m^3/MPa$) for PFA, corresponding to the mix with 380 kg/m^3 binder content and w/b of 0.35, and 51.2 ($MJ/m^3/MPa$) for LC2, corresponding to the mix with 310 kg/m^3 binder content and w/b of 0.35. The energy intensity reduces when it is computed in terms of later-age strength, which is more reasonable for sustainability assessment. Consequently, the values of $ei_{cs,90\ days}$ have been taken for further analysis in the decision framework.

4.4 Sustainability potential of binders through the A-index

Ai_{chlor} is calculated by combining CO_2 emissions with F_{chlor}, which is a durability parameter reflecting the potential for extending the service life of concrete structures under chloride attack. The Ai_{chlor}-value decreases or improves for concretes with lower CO_2 footprint and low chloride penetrability. Ai_{chlor} is plotted against compressive strength at 28 and 90 days in Figure 9. It is seen that, in general, higher strength concretes, having higher amounts of binder, embody larger CO_2 emissions per unit volume, and exhibit marginally higher Ai_{chlor}-values for the same binder type. Ai_{chlor} was highest for OPC mixes, at about 364, while the highest Ai_{chlor} for LC2 and PFA concrete was 236 and 246, respectively. The lowest Ai_{chlor}-value amongst all the concretes reported is 96 for a LC2 concrete, which compares well with the lowest value reported previously for concretes with LC3 [8]. Broadly, the values of Ai_{chlor} are in the order OPC>PFA>LC2, across different strength grades.

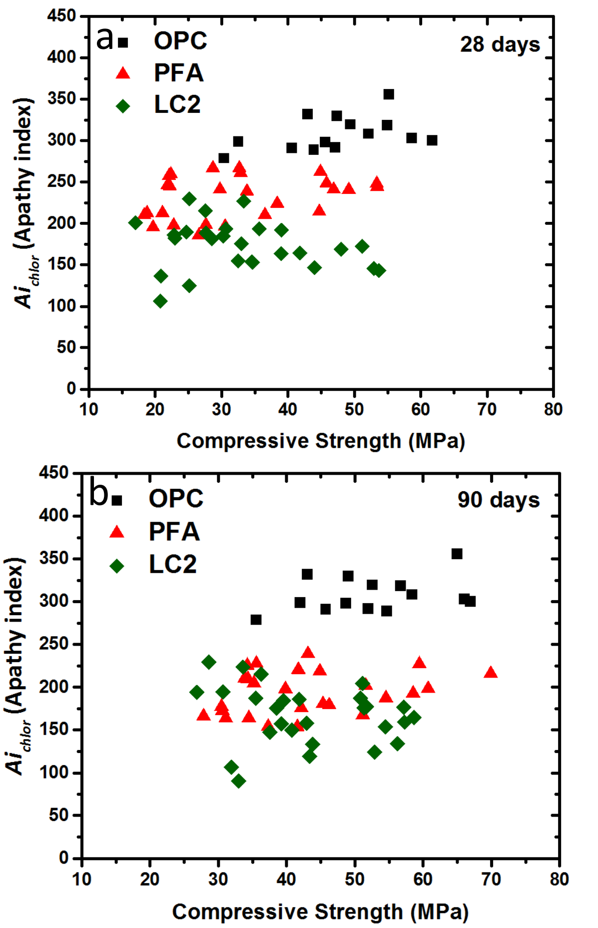


Figure 9. A-indices and compressive strengths of the different concrete systems.

Figure 10 plots Ai_{chlor} and ei_{cs} to present the comparison of sustainability potential for all the concretes. Considering that concretes with lowest Ai_{chlor} and ei_{cs} values have the highest sustainability potential, combining the impacts of CO_2 emissions, concrete durability, energy consumed and strength, the concrete systems lying in the bottom left corner of the plot will be the most sustainable. It is seen that LC2 mixes with high binder content and high w/b yield higher energy intensities due to their low strengths. Such mixes are, therefore, not relevant for practice.

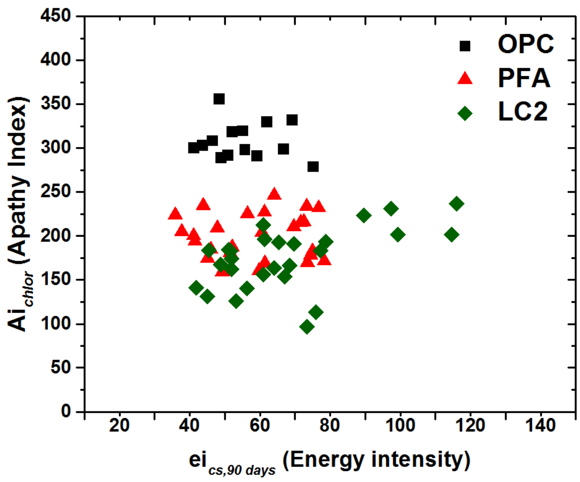


Figure 10. Framework for the choice of sustainable mixes.

In order to assess the influence of strength on the sustainability indices, the data given in Figure 10 is plotted versus strength in Figure 11, considering that strength to be the main basis for specifying concrete for a practical application. In this plot having ei_{cs} on the x-axis, Ai_{chlor} on the y-axis along with 28-day strength on the z-axis, the most sustainable concretes are those closest to the z-axis (marked with red line) for any given strength. The results indicate that OPC concretes are least sustainable, in all the cases considered. Though the sustainability potential is lower for higher strength concretes due to the higher binder contents, it should be noted that, for most structural application, the use of a higher-grade concrete would result in more slender sections, which would reduce the volume of concrete required and the overall impacts.

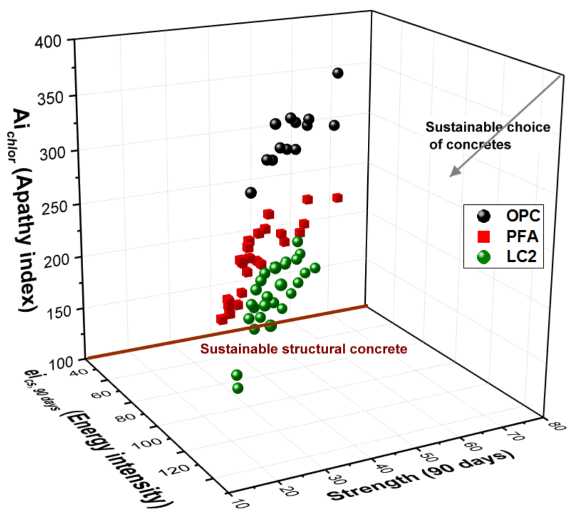


Figure 11. Sustainability framework with the dependence on concrete strength.

5 Conclusions

The potential of limestone calcined clay (LC2) as an admixture for concrete applications has been investigated and benchmarked with OPC and PFA concretes. Based on the systematic approach presented in this study, LC2 is found to be viable as a SCM for a wide range of concrete grades.

The specific conclusions from this study are:

1) The incorporation of LC2 at 45% clinker replacement can yield a wide range of moderate to high strength concretes using conventional mixture proportioning schemes.
2) Significant strength is observed at early ages in LC2 systems with a higher rate of strength development than PFA concretes.
3) The significance of the binder composition on concrete resistivity is reflected by the major increase in the values for LC2 between 3 and 7 days whereas fly ash concretes only exhibit a similar increase in resistivity between 28 and 90 days.
4) Results from the LCA reiterate that concretes with lower clinker content have a positive impact on CO_2 emissions and energy. Though the embodied energy of LC2 is higher than PFA, the carbon footprint is significantly lower for the LC2 concretes than OPC concretes.
5) The sustainability framework used here shows that both LC2 and PFA concretes have higher potential than OPC. Among the concretes studied here, LC2 concretes with w/b ≤ 0.45 and binder content lower than 400 kg/m^3 have the highest sustainability potential.

6 Acknowledgements

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7 Conflict of Interest

Authors declare that there is no conflict of interest

8 Authorship statement (CRediT)

Anusha S. Basavaraj: Conceptualization, Methodology, Software, Validation, Formal analysis, Investigation, Data curation, Writing original draft, Visualization, Writing – review and editing

Hareesh Muni: Data curation, Investigation, Validation

Yuvaraj Dhandapani: Conceptualization, Methodology, Validation, Formal analysis, Investigation, Data curation, Writing original draft, Visualization, Writing – review and editing

Ravindra Gettu: Resources, Writing – review and editing, Supervision, Project administration, Funding acquisition

Manu Santhanam: Resources, Writing – review and editing, Supervision, Project administration, Funding acquisition

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